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UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service
Plant Pest Control Branch
Washington 25, D. C.

TEST METHODS FOR GRANULATED DIELDRIN INSECTICIDE

1. TEST PROCEDURES

1.1 HEOD content. The specific method for HEOD determination under this specification is that of partition chromatography, by the procedure described herein under the title Chromatographic Method. Where the composition is not in question except in regard to quantitative check, the HEOD content may be calculated from determination of the organically bound chlorine, according to the procedure indicated herein under the title Total Chlorine Method 1.1.2.

1.1.1 Chromatographic Method.

Reagents:

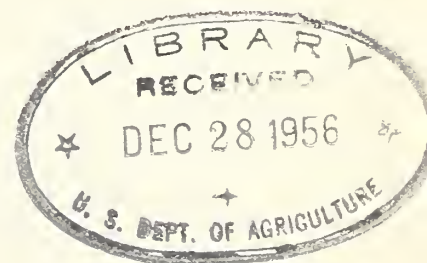
Silicic acid, Mallinckrodt's chromatographic grade
Ethyl ether
N-Hexane, technical, redistilled
Nitromethane, redistilled, colorless
Petroleum ether (Skelly solve F)
Mobile solvent: n-hexane saturated with nitromethane
Mixed dye solution: Dissolve 25 mg. each of D & C Violet No. 2 (1-hydroxy-4-p-toluino-anthraquinone) and D & C Red No. 18 (1-xylylazoxylaze-2-naphthol) in 50 ml. of mobile solvent. Store in a glass-stoppered bottle.

Colorless nitromethane may be prepared by azeotropic distillation with n-hexane. This is conveniently done with a liquid-liquid extractor (Ace Glass Co., Cat. No. 6840 or equivalent). The nitromethane is placed in the boiling flask. The extractor bulb is then charged with n-hexane until it flows back into the boiling flask and covers the nitromethane. Azeotropic distillation is then carried out from the boiling flask. The colorless nitromethane collects as the bottom layer in the extractor bulb.

D & C Violet No. 2 and D & C Red No. 18 are designations of the U. S. Food and Drug Administration.

Apparatus:

Waring Blender with 1-quart mixing cup
Erlenmeyer flasks, 125-ml. - several
Graduated cylinders, two 10-ml., or a two-compartment fraction collector having a capacity of 10 ml. in each arm
Volumetric flasks, several 50-ml. glass-stoppered



Pipets, several 10-ml. volumetric
Chromatographic tube, Scientific Glass Apparatus Co., J-1660-4
Vacuum pump, such as Cenco Pressovac 4
Pressure gage which registers up to 15 lbs. per square inch gage
Pressure pump or compressed air line; if not available use tank
of nitrogen
Hot water bath for evaporating solvent
Soxhlet or Goldfish extractors

Preparation of sample:

Weigh out a sample sufficient to contain 0.75 gram of dieldrin (not HEOD) on the basis of the nominal composition. Extract on Soxhlet or Goldfish with ethyl ether for six hours or overnight. Evaporate the extract until the ether odor is not detected. Transfer to 50-ml. volumetric flask with mobile solvent. Add 2 ml. of the mixed dye solution and make to volume.

Preparation of column:

Use 75 ($\frac{1}{2}$ 0.5) grams of silicic acid, about 42 ml. of nitromethane and 225 ml. of mobile solvent. As each batch of silicic acid is different, the amount of nitromethane required must be determined by trial. Transfer to a Waring Blendor and mix for 15 seconds, then pour mixture quickly into the chromatographic tube. A 3-ft. glass stirring rod worked around in the tube will remove trapped air bubbles. Apply pressure to the column until the silicic acid ceases to settle. Then (optional) release the pressure and remove all except 1 inch of the upper solvent layer using steady suction and a trap to catch the solvent. Apply pressure, up to 8 pounds per square inch, to the column until about 1/8 inch of mobile solvent remains above the silicic acid. (The silicic acid should remain covered with mobile solvent; if the top of the column becomes dry, channeling may occur.)

Operation of column:

Pipet 10 ml. of the prepared sample solution and allow to flow slowly down the side of the tube. Force solution into the silicic acid. Wash wall with 3-5 ml. of mobile solvent, pressing the washing into the column, then gently fill the tube up to within 1 inch of the top with mobile solvent. Apply pressure of 5-8 pounds per square inch.

Collection of fractions:

The HEOD is above the red band, between that and the violet band. When just a trace of the red band still remains above the fritted disc, start taking fractions for HEOD recovery, evaporating each fraction immediately. Take 5-ml. fractions at first, until HEOD appears, then take 10-ml. fractions until end of yield, which should come well in advance of the

violet band. (Caution: The cessation of the appearance of crystalline material, marking the end of the HEOD yield, should be followed by at least one blank flask.) Evaporation of fractions must be done carefully to avoid splattering. A steam bath may be used without suction, or a water bath at 65°C and reduced pressure may be used if the vacuum is controlled by a notched stopper. Combine fractions in a weighed flask; evaporate on a water bath at 60-65°C, using reduced pressure cautiously, and finally evaporate at room temperature with a high-vacuum pump. Weigh the crystalline HEOD residue and calculate the percentage of HEOD in the sample.

1.1.2 Total Chlorine Method. Follow the Total Benzene-Soluble Chlorine Method as given for DDT, in Official Methods of Analysis of the Association of Official Agricultural Chemists. (8th Edition, 1955. Pp. 79-81)

1.2 Grain size distribution.

1.2.1 Sieves. Sieves shall conform to the requirements of specification RR-S-366.

1.2.2 Procedure. Screen a 20-gram sample through a nest of 8-inch U. S. Standard sieves of the designated mesh sizes, using a single-eccentric type mechanical shaker which imparts to the sieve a rotary motion and tapping action of uniform speed of approximately 300 gyrations and approximately 150 taps per minute. Continue the screening for 15 minutes. Weigh the residues and calculate the percentage passing through each sieve. In the weighing and transferring of the sieve sample and its size fractions, any dust clinging to sieve cloth, sieve wall, scale pan and miscellaneous surfaces, or clogging the sieve-cloth apertures, is to be regarded as part of the finest fraction (sieve-pan fraction). Neither this dust nor the material actually entering the sieve pan need be recovered for weighing; the granules lying on the respective sieve cloths are to be recovered and weighed, and the pan fraction calculated by difference.

2. APPLICABLE SPECIFICATIONS AND OTHER PUBLICATIONS

2.1 The following Federal Specifications of the issue in effect on date of invitation for bids, form a part of this specification:

RR-S-366 Sieves; Standard, Testing.

VV-L-791 Lubricants, Liquid Fuels, and related Products; Method of Sampling and Testing.

(Copies of Federal Specifications may be obtained from the General Services Administration, Business Service Center, Region 3, Seventh & D Streets, S. W., Washington 25, D. C. Prices upon application.)

2.2 Other Publications. The following publications, of the issue in effect on the date of invitation for bids, form a part of this specification:

"Official Methods of Analysis of the Association of Official Agricultural Chemists." (Published by the Association of Official Agricultural Chemists, P. O. Box 540, Benjamin Franklin Station, Washington 4, D. C.)

Federal Insecticide, Fungicide, and Rodenticide Act, with amendments. (Copies obtainable from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Prices upon application.)

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Revision of Proposed Federal Specification

Revised by ^DMethods Improvement Section
(with assistance of Dr. H. L. Haller)

Plant Pest Control Branch, ARS, USDA

June 12, 1956

